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A QUANTITATIVE STUDY OF THE EVOLUTION OF GASES FROM ELECTRON TUBES AND MATERIALS

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ABSTRACT

The following is the fourth quarterly report of work aimed at achieving a higher degree of reliability in electron tubes through a better understanding of the kinds and quantities of gases which occur in these tubes. A bakeable, ultra-clean high vacuum system which incorporates an omegatron mass spectrometer has been constructed for conducting these investigations. During this period, several Amperex Type 5894 tubes have been degassed by various schedules. The pumping speed of the ion pump was estimated for methane gas. Several difficulties with heating and operating the tube under test were resolved. The principal gas evolved during DC bombardment of this tube is hydrogen.

INTRODUCTION

This is the Fourth Quarterly Report in the Quantitative Study of Gas Evolution from Electron Tubes. The aims of this project are:

1. To design, construct, and calibrate a system for measuring both the type of gases and the quantities of each type evolved from completely assembled electron tubes.

2. To measure the gases evolved from a suitable electron tube type as a function of its construction materials and of its processing during exhaust.

The effort during the first three quarters has been directed toward the design, construction and checkout of the system. Preliminary degassing and analysis of the Amperex Type 5894 tube was initiated during the Third Quarter. Deuterium gas was admitted to the system during tube degassing to serve as an independent check on throughput.

Variations in the degassing schedule were made with this first tube to determine suitable operating procedures and to explore further the capabilities of the system. These exploratory tests have been partially completed during the Fourth Quarter and two additional tubes have been completely processed while a fourth tube is currently being degassed. An attempt was made to determine the pump speed for methane.

Radio frequency heating of the non-cylindrical anodes of the 5894 proved more difficult than anticipated. A number of modifications in the RF coils and circuitry did not increase the coupling sufficiently to raise the anodes to red heat. DC bombardment of the plate by drawing high plate and screen grid currents proved to be the most effective method of heating these elements. Hydrogen is the gas most copiously evolved during DC bombardment of this tube.

THE TUBE UNDER TEST

The tube type chosen for this study of total gas evolution is Amperex Type 5894. This tube is in volume production and under good control with a yield in excess of 90%. It is a twin, four-electrode tube designed for use as a radio frequency power amplifier, oscillator, modulator and frequency multiplier. Each anode is capable of dissipating 20 watts. A single, indirectly heated, oxide-coated cathode is used. The materials of construction are listed below:

1. Glass: Corning 7052 or equivalent.
2. Cathode: Nickel.
3. Beam Plate: Double carbonized nickel.
4. Screen Grid: Molybdenum and nickel side rods, molybdenum laterals. Entire grid coated with aquadag.
5. Control Grid: Molybdenum and nickel side rods, gold plated molybdenum laterals.
6. Cathode Coating: Barium and strontium carbonates.
7. Filament: Tungsten with aluminum oxide insulation.
8. Spacers: Mica, two with magnesia coating and two without.
9. Anodes: Molybdenum with zirconium.
10. Miscellaneous pins, connectors and straps: Kovar, molybdenum and nickel.
11. Getter: Barium, aluminum and iron.

Since we planned to use completely assembled but otherwise unprocessed tubes in our experimental work, we attempted to estimate the gas load to be handled by the system. Based on information from the literature we expected the total gas from the metal parts to be in the neighborhood of a few hundred liter microns. The major contributor of the total gas to be removed will be due to the carbon dioxide released by the cathode coating during the calcining operation (carbonate breakdown). Assuming the barium-strontium carbonate is equimolar and knowing that the quantity of coating is about 20 mg., we can calculate the amount of carbon dioxide released during breakdown. The amount of coating binder is negligible and can be ignored.

Using the equation

$$BaCO_3 \cdot SrCO_3 + \text{Heat} \rightarrow BaO \cdot SrO + 2CO_2$$

then 20 mg. of coating would result in 2160 micron liters of CO_2

Because the sensitivity of the omegatron decreases rapidly with increasing pressure in the neighborhood of 10^{-5} Torr, the maximum throughput is determined by this pressure limitation. Limiting the omegatron pressure to about 8×10^{-6} Torr would limit the total throughput of CO_2 to 0.034 micron liters/second (Figure 1) or 2.04 micron liters/minute. At this rate it would require 1060 minutes or about 18 hours to remove the carbon dioxide provided, of course, that this was the only gas present.

TOTAL GAS RATE VS. OMEGATRON PRESSURE

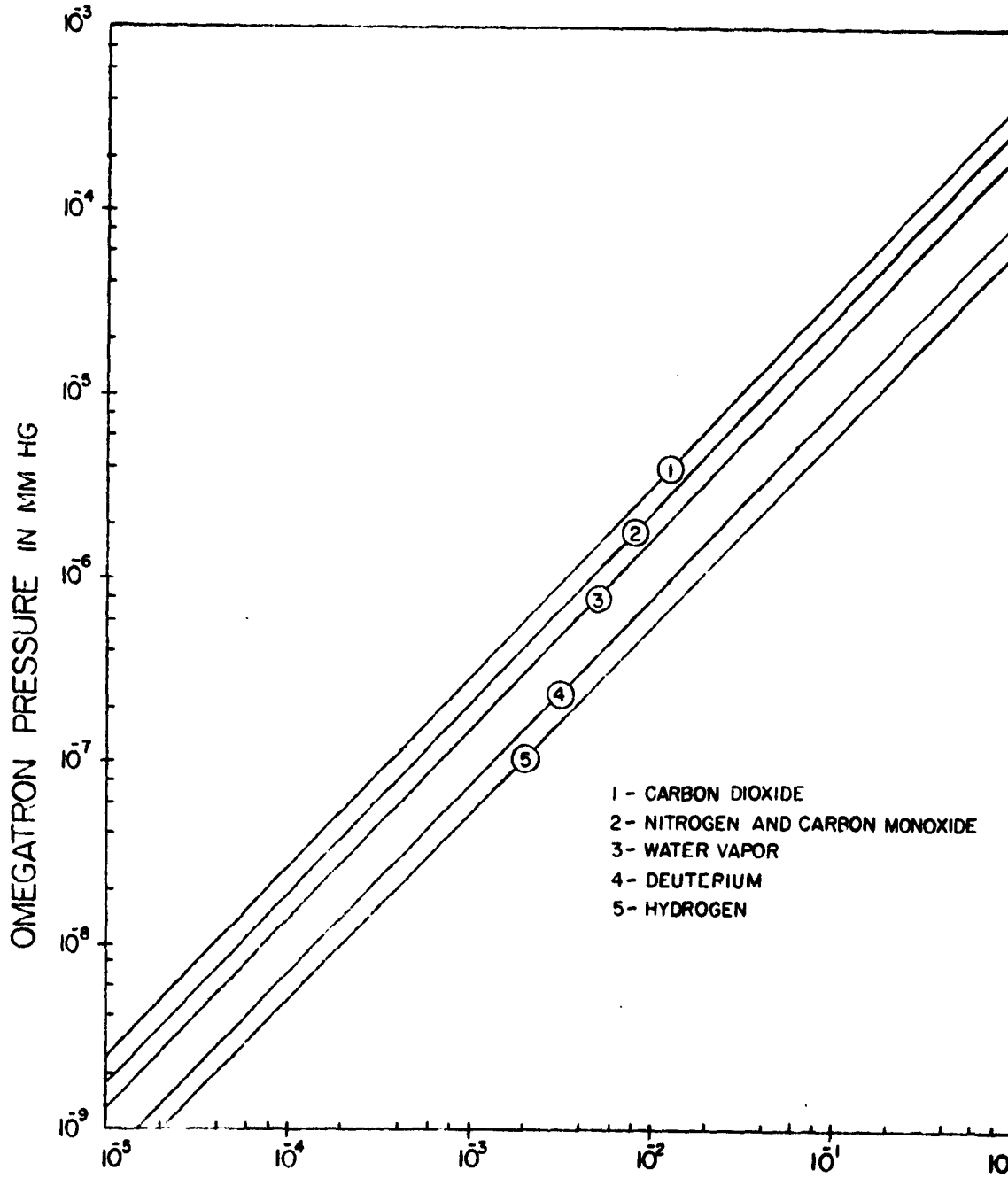

FIG. 1

USE OF DEUTERIUM AS A TRACER GAS

In the Third Quarterly Report mention was made of the attempt to use deuterium (Symbol D_2, mass=4) as an independent check on the omegatron throughput. A relatively large peak appeared at the mass to charge ratio of 20 upon admitting the D_2 to the system. Since there was no peak at mass 40, the peak at mass 20 could not be doubly ionized argon. It was therefore persumed to be D_2O. Close scrutiny of the variations in the peak heights at mass 4 and 20 revealed no satisfactory correlation between these peak heights and the changes in RF heating of the tube. The D_2O peak at mass 20, then, is probably a result of reactions at the filaments of the ion gauge or omegatron between D_2, CO and CO_2. The following partial pressures existed at the omegatron:

CO_2:	1.52×10^{-8} mm Hg.
N_2:	4.52×10^{-8} " "
CO:	1.03×10^{-8} " "
D_2:	3.12×10^{-7} " "

Hence, the throughput of D_2 was 4.8×10^{-3} micron liters/second (From Figure 1). This rate remained constant throughout this period. The throughputs of the other gases ranged from 8.6×10^{-5} micron-liter/second to 7.8×10^{-3} micron liters/second. In Table 1. are shown the peak heights in millivolts (across the 10^{10} ohm resistor) together with pressure reading at P_2 (See Figure No. 2) ion gauge. The values for D_2, CO_2, CO and N_2 have been converted to throughputs and are shown in Figures 3 and 4.

Preliminary Degassing of Tube No. 1.

Time	P_2 mm.	Mass Number							
Min.	($X10^4$)	4	12	14	16	18	20	29	44	Remarks
2 1/2	2		.6	.6	1.8	1.2	3.3	27	6.4	Rf on at 0 time
6	1.2		.6	.75	1.8	1.2	3.6	58	15.9	
9	.3		.2	.4	.2	1.1	2.8	6.9	1.2	RF off then on
16	.6	12	.8	.7	1.0	1.0	2.8	21	3.2	
19	4.2		.6	.6	1.0	1.2	2.8	28	6.0	RF increased
22	4.		1.0	.8	1.2	1.1	2.8	28	7.2	
25	3.5		.7	.6	1.0	.9	2.8	26	7.8	
27	.15									RF off
31	.2					1.8	4.5	9.0	3.0	RF on
33	.32		.6	.6	.63	1.2	4.5	15.0	5.0	
37	.35	12	.7	.7	1.0	1.6	4.2	21.0	7.6	
41	.35		.8	.6	1.2	1.4	4.3	23.0	7.8	
44	1.3			.75	3.0	1.5	4.2	50.0	9.9	RF increased
47	1.3		3.0	1.05	3.9	1.8	4.5	100.0	30	
51	1.1		2.7	.8	3.6	1.5	5.0	84.0	32	
55	1.		1.3	.8	3.3	1.6	4.9	75.0	28	
66	.08		2.1	.9	2.6	1.8	5.3	60.0	25	
75			1.8	.8	2.5	1.7	5.2			
86	.06		1.2	.7	1.8	1.7	5.2	40.0	15	

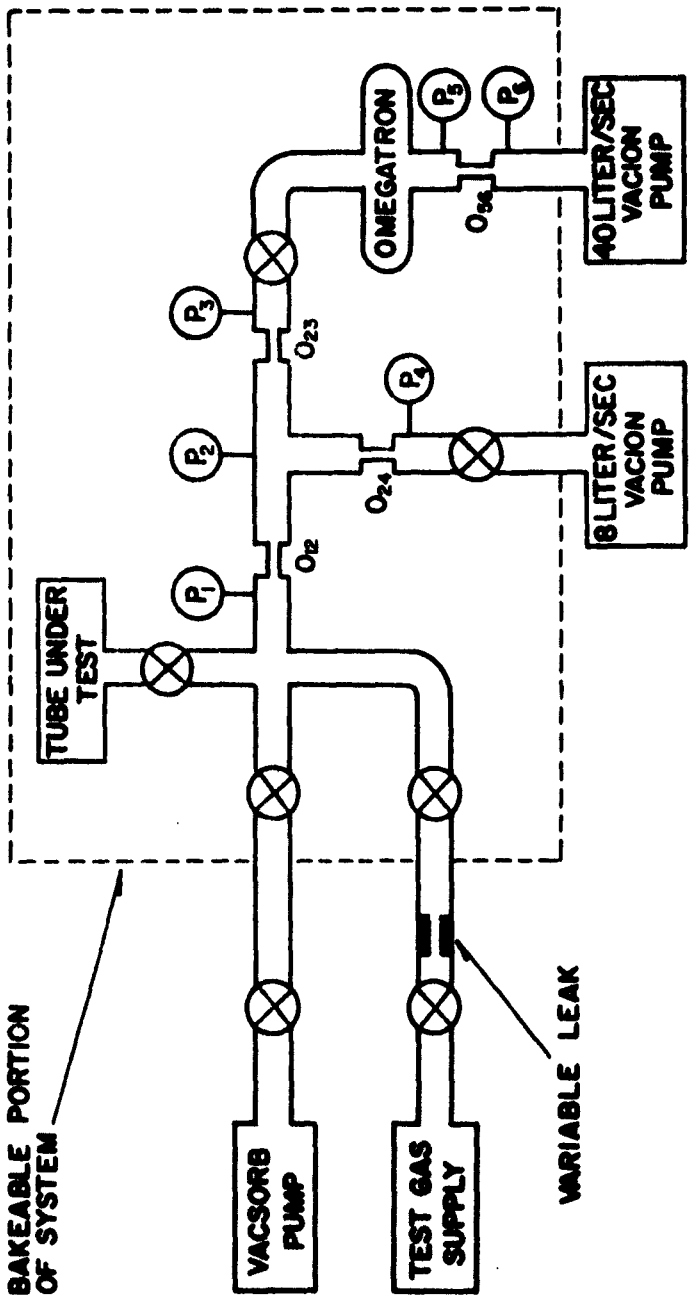
BAKEABLE PORTION OF SYSTEM
TUBE UNDER TEST
VACSORB PUMP
TEST GAS SUPPLY
VARIABLE LEAK
OMEGATRON
40 LITER/SEC VACION PUMP
8 LITER/SEC VACION PUMP
P1
P2
P3
P4
P5
O12
O23
O24
O34

Figure 2

INDIVIDUAL THROUGHPUT vs. TIME
(SHOWING D_2 TRACER GAS)

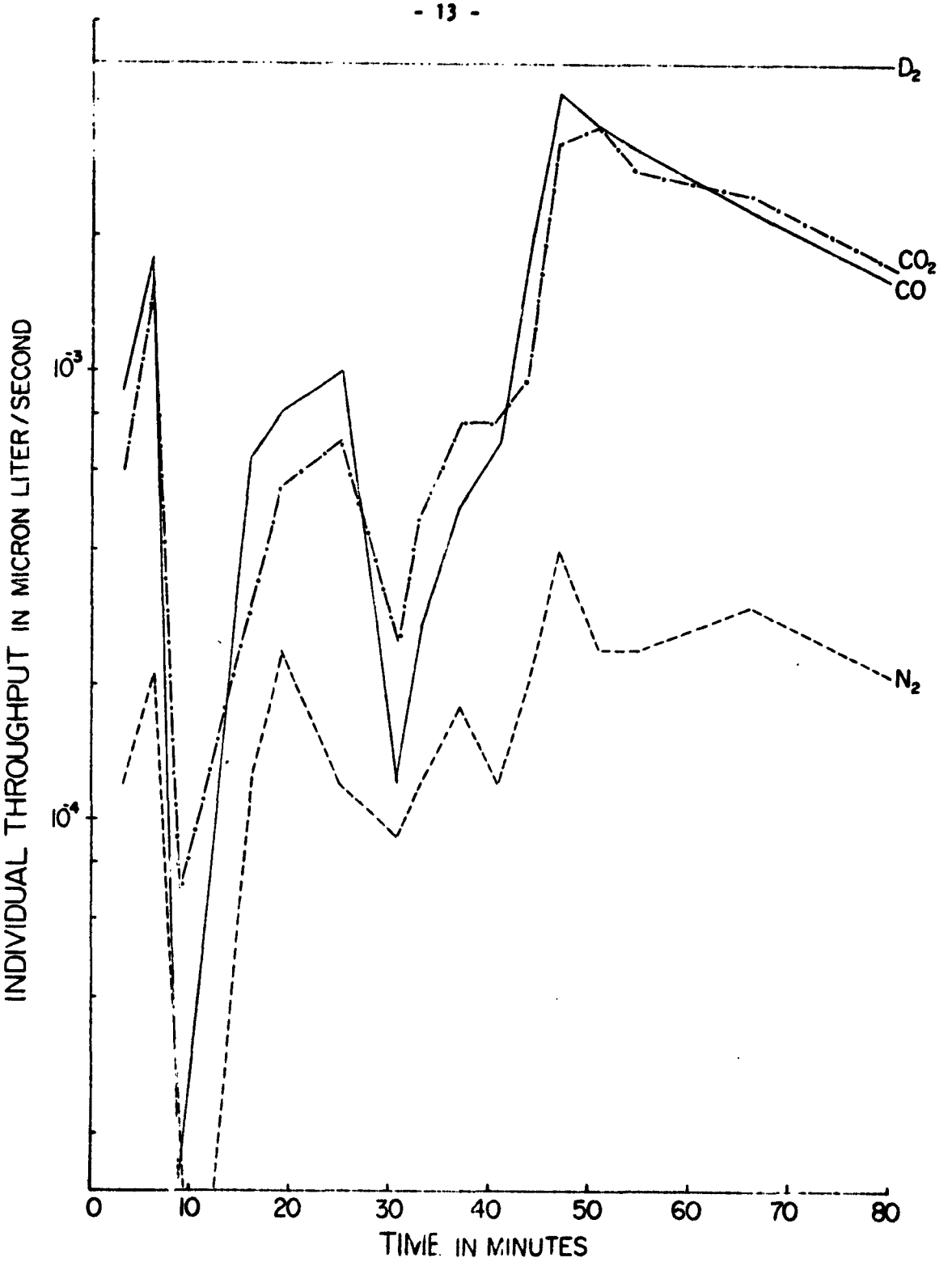

FIG. 3

TOTAL THROUGHPUT vs. TIME
(SHOWING D_2 TRACER GAS)

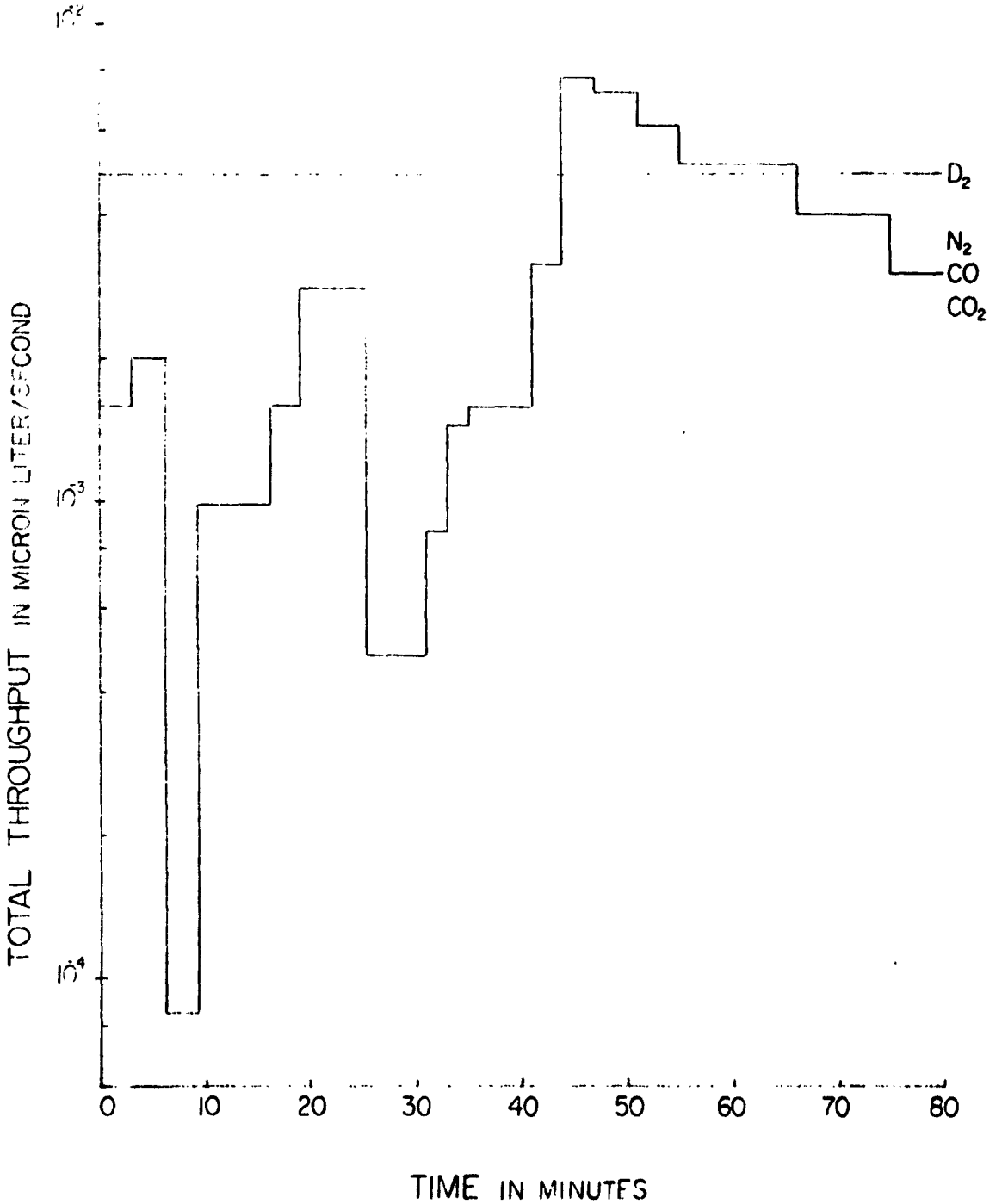

FIG. 4

ESTIMATION OF PUMP SPEED FOR METHANE

An attempt was made to estimate the pump speed for methane since this was not known. To do so, methane was admitted through the variable leak. Pressure readings were taken at gauges P_5 and P_6 across the orifice O_{56}. The omegatron was scanned and the pressure, P_{40}, read from the 40 liter per second pump.

CH_4 Cylinder Pressure	P_5	P_6	P_{40}
55PSIA	1.4×10^{-6}	6.5×10^{-8}	1.2×10^{-8}

The omegatron scan revealed the presence of CO at 2×10^{-7} mm Hg as well as CH_4 at 1.3×10^{-6} mmHg. The amount of CO is approximately 13.3% of the total CO + CH_4. We assume that the composition of the gas is the same at the ion pump, P_{40}. The gauge reading at P_5 can be expressed as the sum of the hypothetical gauge readings due to CH_4 and CO:

$$R_{CH_4} + R_{CO} = 1.4 \times 10^{-6}$$

where R is the hypothetical gauge reading. Substituting for the R values the actual value (A) multiplied by the ratio of the ion gauge sensitivity for the particular gas to that for nitrogen we obtain the equation:

$$13.92\ A_{CH_4}/10 + 10.38\ A_{CO}/10 = 1.4 \times 10^{-6}$$

Since

$$0.133\ A_{CH_4} = A_{CO}$$

we have

$$A_{CH_4} = 9.15 \times 10^{-7} \text{ mm Hg}$$

Similarly for P_6:

$$17.74\, A_{CH_4}/10 + 10.42\, A_{CO}/10 = 7.6 \times 10^{-8}$$

or

$$A_{CH_4} = 3.98 \times 10^{-8} \text{ mm Hg}$$

To calculate the pump speed for methane, we use the relation:

$$S_{40}\, P_{40} = C_{56}\, (P_5 - P_6)$$

where: S_{40} is the speed of the 40 liter per second pump;

P_{40} is the pressure of the 40 liter per second pump;

C_{56} is the conductance of the orifice between P_5 and P_6.

For methane:

$$C_{56} = 0.85 \text{ liters per second}$$

The value of P_{40} for CH_4 alone we determine from the ratio of total pressure at P_6 to P_{40}. The average of ten readings taken during the nitrogen calibration gives a value of 3.38 for P_6/P_{40}. Then P_{40} for CH_4 is 1.18×10^{-8}. Therefore, S_{40} may be calculated as 63 liters per second for methane.

From Figure 6 of the Third Quarterly Report, "Ion Pump Speed vs. Pressure", the speed of the 40 liter per second pump at 1.2×10^{-8} mm Hg is about 31 liters per second. Based upon this admittedly rough estimate (of pump speed for methane), the methane pumping speed is about twice that for nitrogen. This result is not too discrepant from the manufacturers literature which states that the pump speed for hydrocarbons ranges from 0.9 to 1.6 times that for nitrogen.

PROCEDURE FOR DEGASSING THE TUBE UNDER TEST

In our intended procedure for degassing the tube under test we planned to proceed as follows:

1. Attach assembled but unprocessed tube to system.
2. Bake tube and system overnight at 450°C.
3. Scan mass range 2 to 44 to determine any background pressures.
4. Heat the tube under test using RF induction heating so as to maintain a fairly constant degassing rate.
5. Scan the mass range 2 to 44 every five minutes.
6. After a period of time, light the filament of the TUT and proceed to calcine (breakdown) the carbonate coating of the cathode at the same time varying or eliminating RF heating to test the effects of such variations.
7. Apply voltages to the plates and grids of the TUT to draw current to these elements and heat them by DC bombardment.
8. Calculate the partial pressure of each gas for each scan.
9. Determine the degassing rate of each gas using Figure 1.
10. Compute the total gas evolved by multiplying the rate of degassing by the time over which it occurred.

Some difficulties and unexpected factors, however, necessitated several changes in the general plan. Foremost of the difficulties was that of adequately heating the plates of the TUT. These plates are essentially flat with no circular connecting path, a configuration extremely difficult to heat by RF induction. Despite construction of a number of helical and ear-muff coils, we were unable to couple effectively enough to raise the plates to red heat. The cathode and grids, on the other hand, attained

700° - 800°C rather easily with the helical coil. Maximum output of the 3 KW radio frequency generator, however, still could not heat the plates as desired.

Another unexpected factor was the length of time required for completing the cathode breakdown. As shown earlier, the approximate amount of CO_2 released by the carbonate coating is 2160 micron liters. This represents a considerable quantity of coating in itself. The estimated time required for removal of the CO_2 by our system is almost 18 hours. We attempted to monitor the cathode breakdown on Tube #2. After 65 hours (Scan 32A) the mass 44 peak (CO_2) had dropped considerably but not to the extremely low level anticipated. Furthermore the total pressure indicated at P_1 had not decreased at all. Apparently the composition of the gases coming off the cathode was changing but the total quantity remained as high as before. Table II reports the peak heights obtained for mass-to-charge ratios 12 through 44 (highest m/e number scanned) during cathode breakdown. Figure 5 shows how the CO_2 ion current varied with RF heating and filament voltage during breakdown. Figure 6 is a typical scan made with both the RF heating and filament voltage applied.

In the interest of time, the monitoring attempt was terminated and the breakdown completed using the sorption (VacSorb) pump shown in the block diagram of Figure 2. Following this step, the system was again baked. After cool down and removal of the oven, a small leak was detected in a metal bellows near one of the Granville-Phillips valves. This was repaired after which DC bombardment of the TUT was initiated. Plate

TABLE II. DATA ON CATHODE BREAKDOWN ON TUBE #2

Scan Number	Elapsed Time Minutes	Ef v	RF v	mv at Mass Number 12	14	16	17	18	20	22	28	32	40	44
1	5	0	.5								.99			.09
2	15	0	.5								.99			.09
3	18	0	1.0								1.08			.09
4	23	0	1.0								1.11			.12
5	28	0	1.3	1.8	.09	.18					9.6			.12
6	38	0	1.7	7.6	1.5	3.2	.2	.9			249		.45	.78
7	48	0	1.7	5.3	1.0	2.3	.2	1.0			174		.3	1.2
8	56	0	1.7	5.2	.9	2.0	.2	.9			150		.2	1.3
9	66	0	1.7	3.2	.4	1.4		.6			114			1.4
10	78	0	1.7	2.8	.4	1.2		.4			90			1.8
11	85	0	2.0		3.0	24		4.2		1.2	288			66
12	95	0	1.0			.18		.18			7.2			9.0
13	103	0	1.0	.18		.42		.42			4.8			4.4
14	116	0	1.0	.12		.18		.24			3.0			2.2
15	123	0	1.0	.12		.18		.3		.06	2.8			1.9
16	151	0	1.0	.06		.12	.06	.18			2.0			1.7
17	181	6.0	1.0	15	1.2	19				1.2	282			42
18	188	6.0	1.0	2.4		2.4					30			40
19	192	7.5	1.0	2.4							150			18
20	196	9.0	1.0	7.2	.6	4.2					210			54
21	202	10.5	1.0	13.5	1.8	9.0					246			80
22	217	12.0	1.0	36	5	25		.6			225			180
23	225	12.0	1.0	32	4.2	24		.6		.9	680			190
24	240	12.0	1.0	30	3.0	25		.6	.6	1.2	640			230
25	258	12.0	1.0	24	3.0	22		.4	.2	20	600	.4		230
26	378	0	0	1.0	.1	1.3					7.2	.3		13.6
27	398	0	0								.3			.12
28	414	0	1.0	.54		.3		.06			12			.6
29	423	6.0	1.0	1.5		.72					28			.72
30	427	9.0	1.0	3.4		1.0					130			7.2
31	437	12.0	1.0	28		44					168			240
32														
33	520	12.0	1.0	10.2	.12	16.8				1.2	68	4.4		260
34	552	12.0	1.0	5.2	.8	7.8				.6	24	1.8		126
1A	580	12.0	1.0	3.2		5.6				.2	10.8	.6		50
2A	587	12.0	0	.42	.06	1.0					15.6	.12		9.6
3A	733	0	0	.24		.54					2.0			4.8
4A	788	10.5	0	4.0	.66	.8				.18	92		.18	18
5A	804	10.5	0	5.4	.8	4.8			.06	.54	150			36
6A	812	10.5	0	5.2	.7	5.2			.06	.48	150	1.9		38
7A	852	10.5	0	5.4	.6	5.4			.12	.6	150	.06	.24	46
8A	886	10.5	0	4.8	.4	5.0			.12	.54	126	.12	.18	44
9A	897	10.6	0	5.4	.4	5.8			.12	.54	144	.24	.24	50
10A	977	10.8	0	5.6	.6	7.0			.06	.60	120	.90	.18	66
11A	1018	10.8	0	5.4	.6	7.4			.12		138	1.6	.24	82

TABLE II. DATA ON CATHODE BREAKDOWN ON TUBE #2

Scan Number	Elapsed Time Minutes	Ef v	RF v	12	14	16	17	18	20	22	28	32	40	44
11A	1018	10.8	0	5.4	.6	7.4			.12		138	1.6	.24	82
12A	1054	10.8	0	5.2	.6	7.4			.06	.60	100	1.9	.18	76
13A	1084	10.8	0	5.8	.66	8.0			.18	.72	138	2.4	.30	86
14A	1154	10.8	0	6.0	.6	8.4			.12	.72	120		.24	92
15A	1186	10.8	0	4.6	.36	5.8			.06	.48	90	2.4	.18	72
16A	2086	11.0	0	3.6	.3	5.7				.48	57		.18	60
17A	2090	11.0	0	3.6	.28	5.4			.06	.48	56	3.3	.18	60
18A	2120	11.1	0	4.2	.36	6.2			.06	.48	66	3.8	.18	78
19A	2129	11.1	0	4.8	.36	7.2				.48	68	4.4	.36	90
20A	2176	11.3	0	4.0	.3	5.4				.30	47	3.2	.12	54
21A *	2185	11.3	0	6.0	.66	9.7				.90		6.9		108
22A	2190	11.3	0	5.8	.72	9.0			.12	1.0	114	7.4	.27	120
23A	2232	9.0	0	.66		1.0					10	.78		14
24A	2267	9.0	0	.48		.72				.06	9.0	.54		8.4
25A	2342	9.0	0	.42		.78					8.4	.42		7.2
26A	2418	9.5	0	.48		.78					10.8	.54		9.0
27A	2525	9.5	0	.48		.84					11.4	.54		9.6
28A	2626	9.5	0	.48		.72					9.6	.50		8.4
29A	3291	9.0	0	.48		.78					10.2	.54		9.0
30A	3296	9.0	0	.42		.72				.06	10.2	.48		8.4
31A	3558	9.5	0	.60		1.0				.12	12.0	.60		10.8
32A	3818	9.5	0	.60		.90					12.0	.60		10.2
33A	3823	6.0									.24			

Cathode breakdown completed through Vacsorb Pump.

* Repeak RF Bias

CO_2 EVOLVED DURING CATHODE BREAKDOWN
(EFFECT OF FILAMENT AND RF HEATING - TUBE NO. 2)

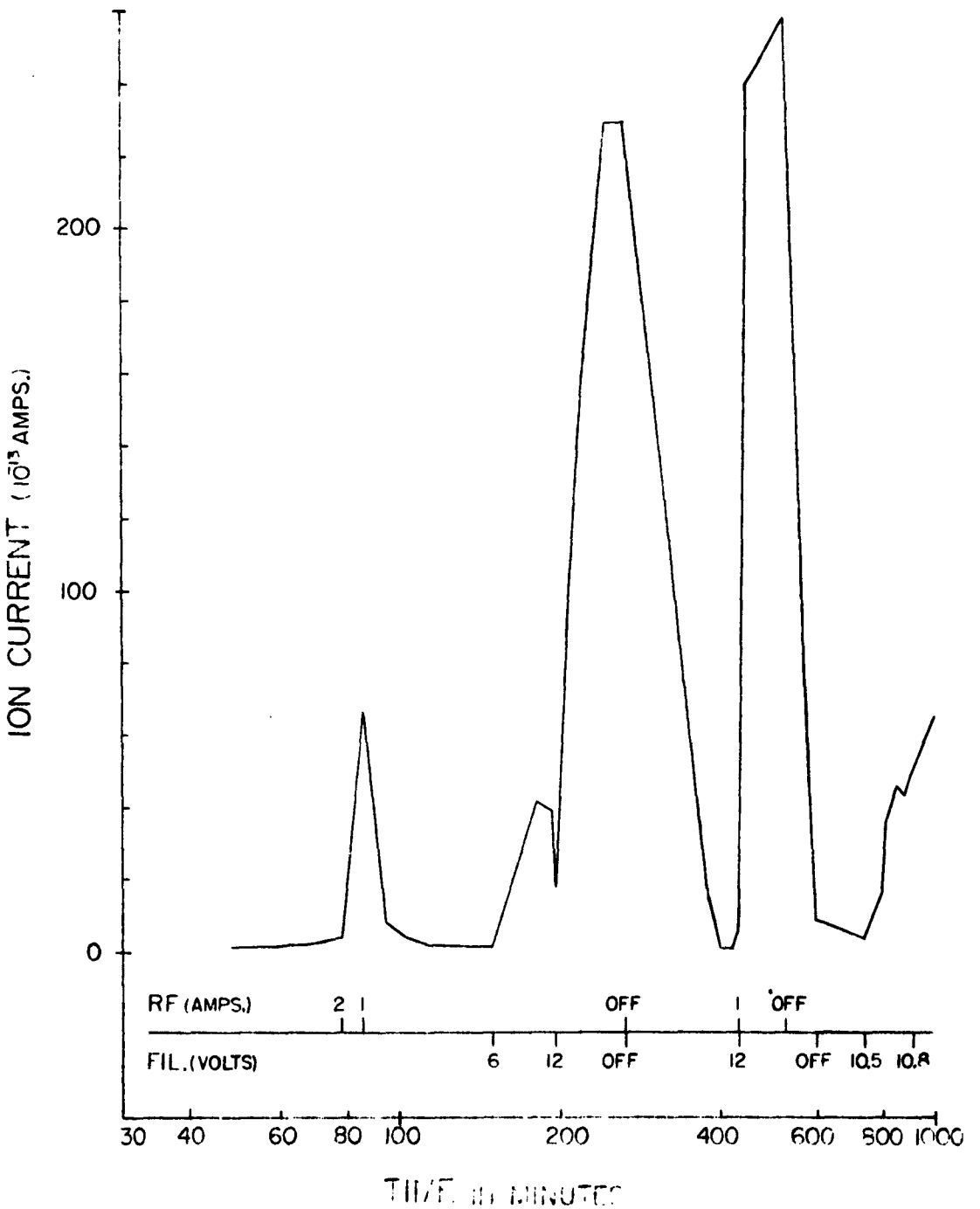

FIG. 5

CATHODE BREAKDOWN
(TUBE NO. 2 – SCAN NO. 31)

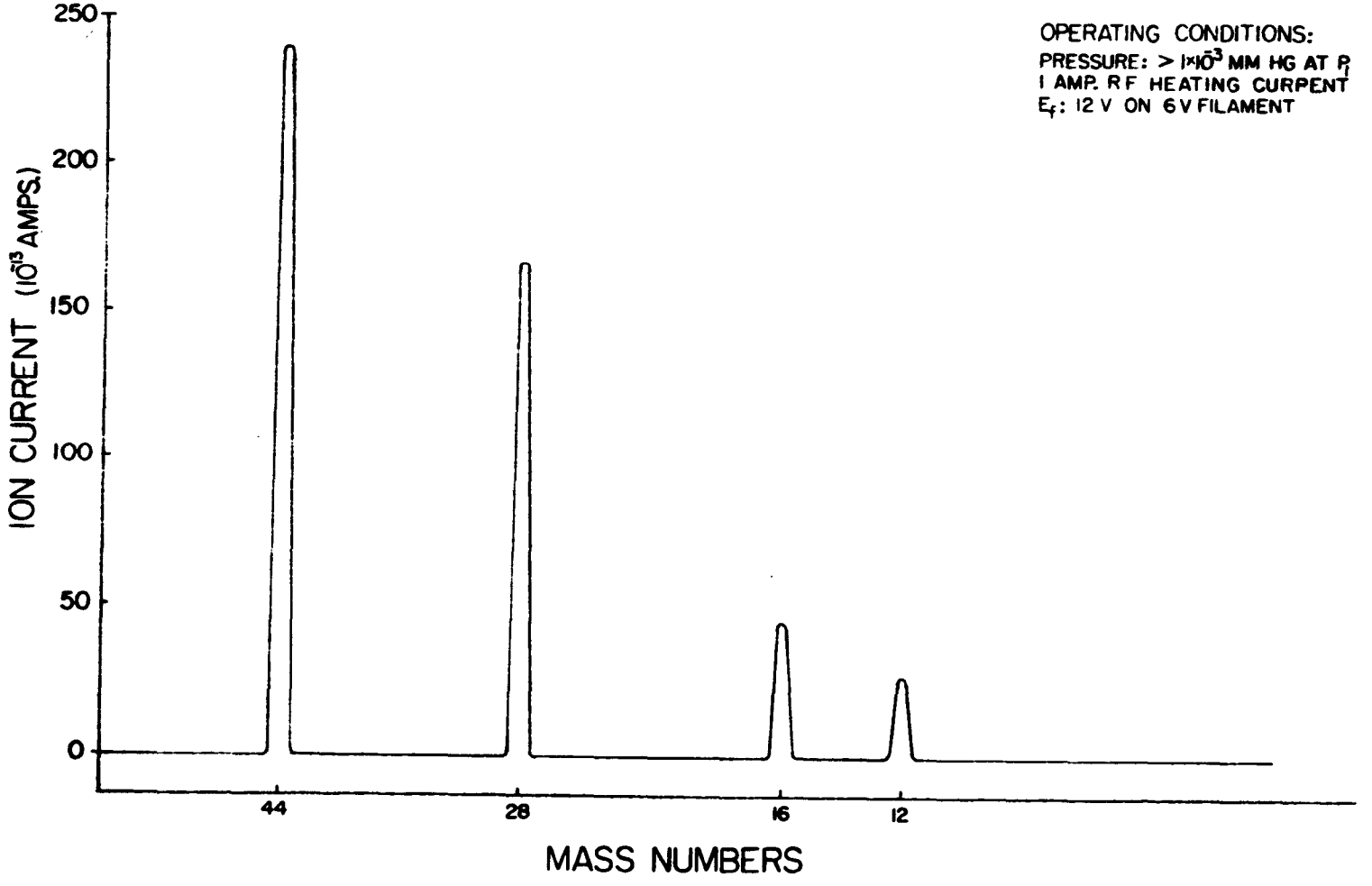

FIG. 6

temperatures of 800° to 900° C were attained by this method. Figures 7 and 8 show the effect of varying plate current (and thereby its temperature) on the degassing rate of hydrogen, the principal gas evolved during DC bombardment of the plate. The ordinate is plotted as ion current to emphasize the effect of changes in plate current on the hydrogen evolved.

Attempts to heat the control grid by applying a small positive voltage to it (in fact, even grounding the grid) caused wildly fluctuating readings in all gauges. Apparently, the additional capacitance of the lead wire connection caused the tube to begin oscillating and interacting with power lines of associated equipment. After some experimentation we were able to prevent oscillation by connecting an air-core inductance at the plate lead in series with the plate current.

The fact that the zirconium coating on the plate is a getter probably provides another difficulty in this degassing study. The barium-aluminum getter was never activated during any experiments or processing treatments. The zirconium gettering may under some circumstances interfere with the degassing measurements by removing a portion of the evolved gas by reaction or absorption.

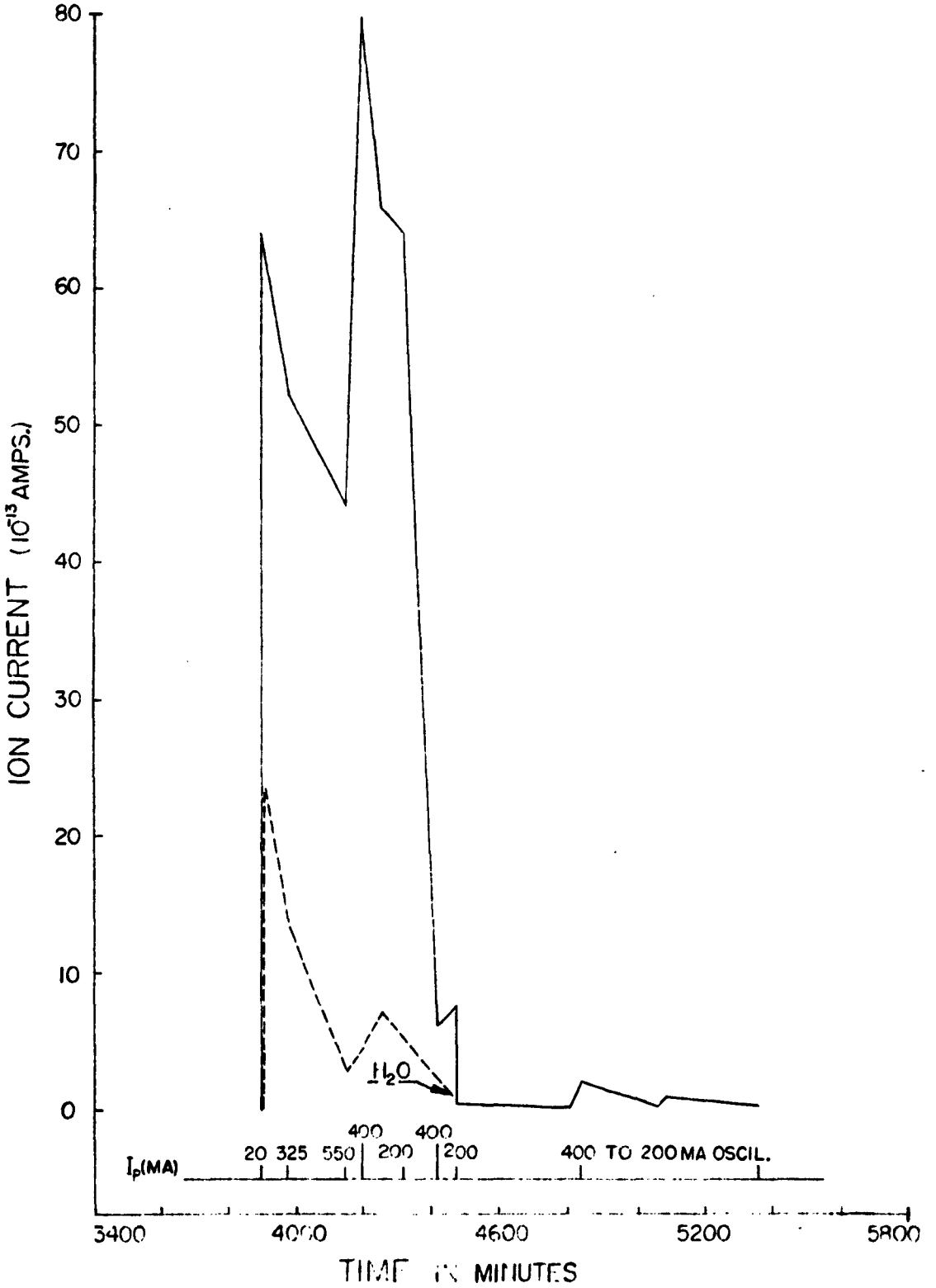
H_2 EVOLVED DURING DC BOMBARDMENT
(TUBE NO. 2)
ION CURRENT (10^{13} AMPS.)
80
70
60
50
40
30
20
10
0
H_2O
400
400
I_p(MA)
20 325 550
200
200
400 TO 200 MA OSCIL.
3400
4000
4600
5200
5800
TIME IN MINUTES

FIG. 7

H_2 DURING DC BOMBARDMENT
(TUBE NO. 3)

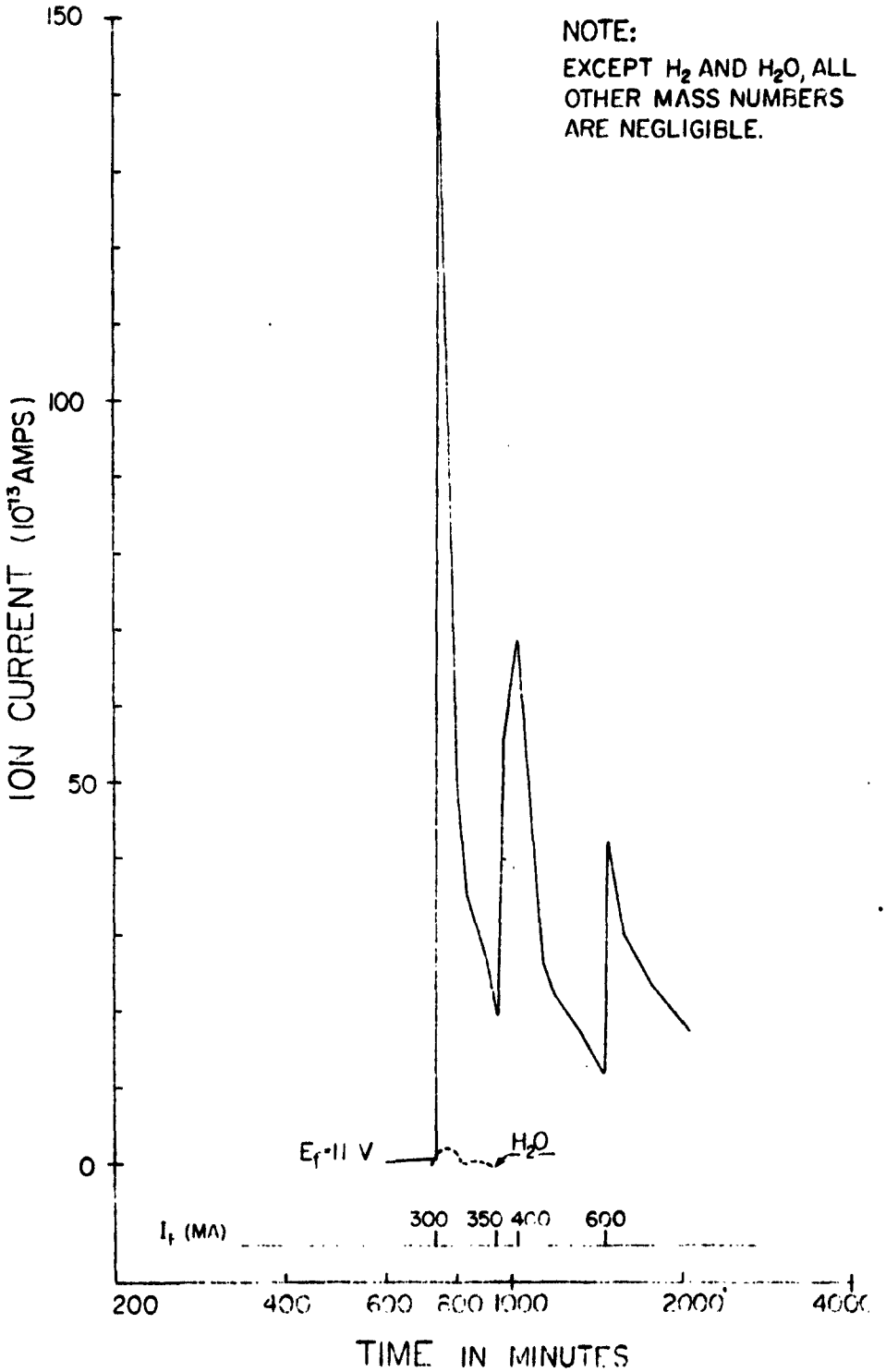

FIG. 8

RESULTS OF TUBES #2 AND #3

Because a great deal of experimentation with degassing conditions and system parameters was performed on Tube #1, no detailed analysis of the data was attempted. Additional experimentation was performed on Tubes 2 and 3 in efforts to determine the effects of various treatments on the amount and composition of gases released by the tube elements. Figure 5 illustrates the effects of RF heating and filament voltage changes on the CO_2 ion current. Typical scans taken during cathode breakdown are shown in Figures 6, 10 and 11. The total pressure indicated at ion gauge P_1 is of the order of one micron in each case (between 7.4×10^{-4} and 1.5×10^{-3} mm Hg). The different patterns obtained with RF heating plus filament voltage (Figure 6), RF heating alone (Figure 10) and filament heating alone (Figure 11) were at first thought to be related specifically to the different operating conditions used in heating the TUT.

As mentioned earlier, the helical RF coil was quite effective in raising the cathode and grids to red heat. Even though it was less effective on the plates, it did heat them somewhat. This RF induced heat undoubtedly caused some desorption of gases from the metal parts of the tube and would account to some extent for the different patterns shown in the three scans (Figures 6, 10 and 11.) The zirconium coating on the plates of the TUT is a good getter, also, a fact which adds further complexity to the situation. Furthermore, other investigators (1, 2) have reported differences in the residual gas content of tubes depending on the combinations of RF heating, filament voltage and oven baking during cathode breakdown.

However, it was not until all the calculations and summations were completed and plotted as shown in Figure 9 that the reason for some of of the anomalous behavior during cathode breakdown became apparent. The specific clue was the unexpectedly high nitrogen (N_2) total. In retrospect, this must have been due to the beginning of the leak in the bellows subsequently detected after the oven baking of the system following breakdown completion with the sorption pump. The leak was so small that it did not overload the ion pumps during the breakdown process but later opened up more due to the bakeout and prevented the pumps from reaching their ultimate pressure. It went undetected as a cause of trouble during breakdown until calculations were complete primarily because both N_2 and CO occur at the m/e ratio of 28. Only by analysis of secondary peaks at other m/e ratios can N_2 and CO be distinguished. Since this (Tube No. 2) was our first attempt at monitoring the cathode breakdown process, extensive analysis was not attempted during the process. Although a slight oxygen (O_2) peak begins to appear at m/e=32 in the course of the breakdown, this was ascribed to possible changes in the process mechanism. Needless to say, future runs will be analyzed more completely while in progress to prevent recurrence of such an event.

Following repair of the leak, DC voltages were applied to the plates and screen grids of the TUT. By DC bombarding these elements, plate temperatures of 800-900°C were achieved. Again, Figures 7 and 8 illustrate the effect of plate current on the ion current of H_2 and H_2O. The time scales on the two figures are not comparable in absolute value because they are referred to in the first omegatron scanning which took place at different times for the two tubes. In the case of Tube No. 2, the first scan was made before cathode breakdown. To conserve time, the

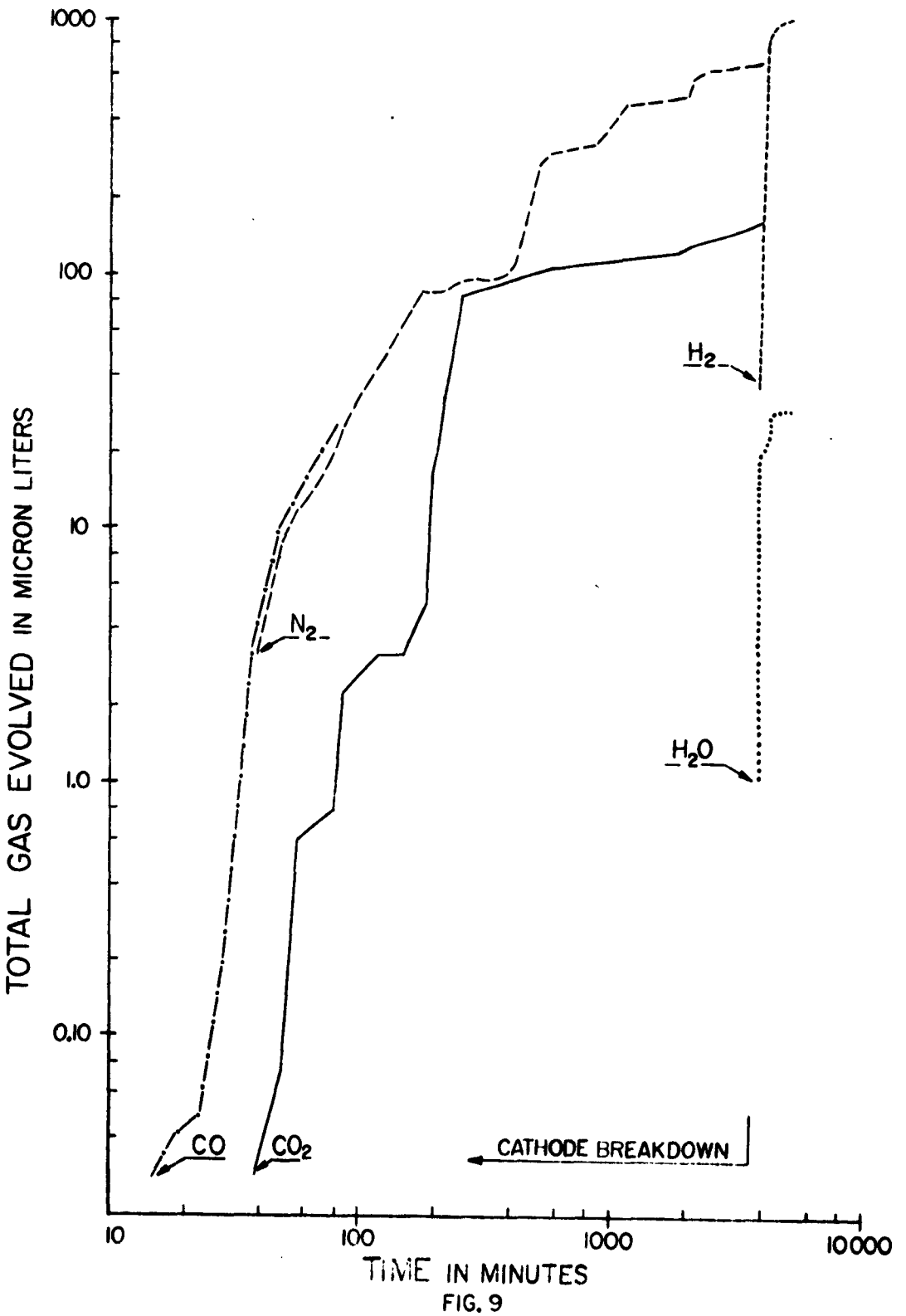
TOTAL GAS EVOLVED (TUBE NO. 2)
TOTAL GAS EVOLVED IN MICRON LITERS
1000
100
10
1.0
0.10
H_2
N_2
H_2O
CO
CO_2
CATHODE BREAKDOWN
10
100
1000
10000
TIME IN MINUTES

FIG. 9

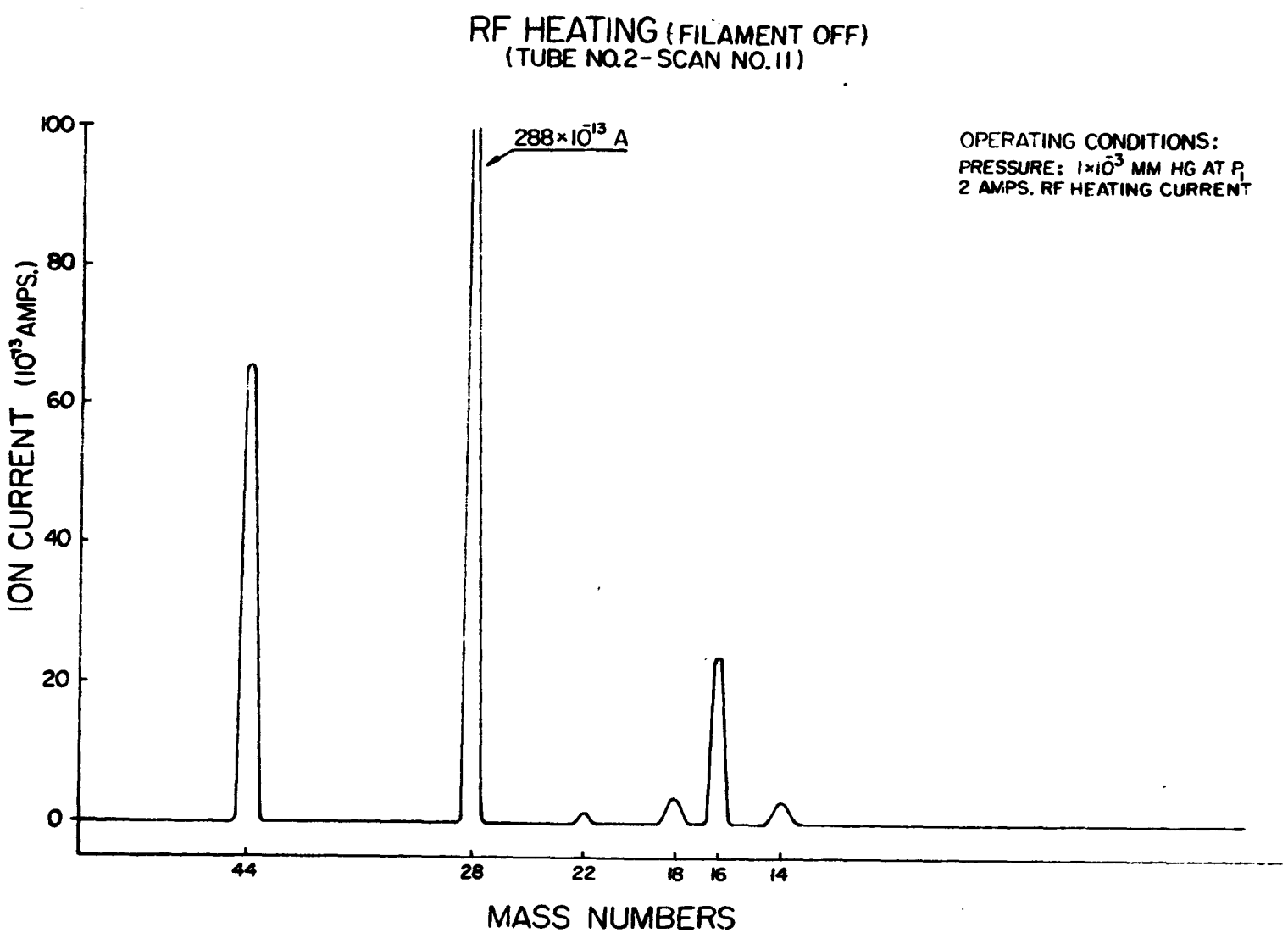
RF HEATING (FILAMENT OFF)
(TUBE NO.2 - SCAN NO.11)
288×10⁻¹³ A
OPERATING CONDITIONS:
PRESSURE: 1×10⁻³ MM HG AT P₁
2 AMPS. RF HEATING CURRENT
ION CURRENT (10⁻¹³ AMPS.)
100
80
60
40
20
0
44
28
22
18
16
14
MASS NUMBERS

FIG. 10

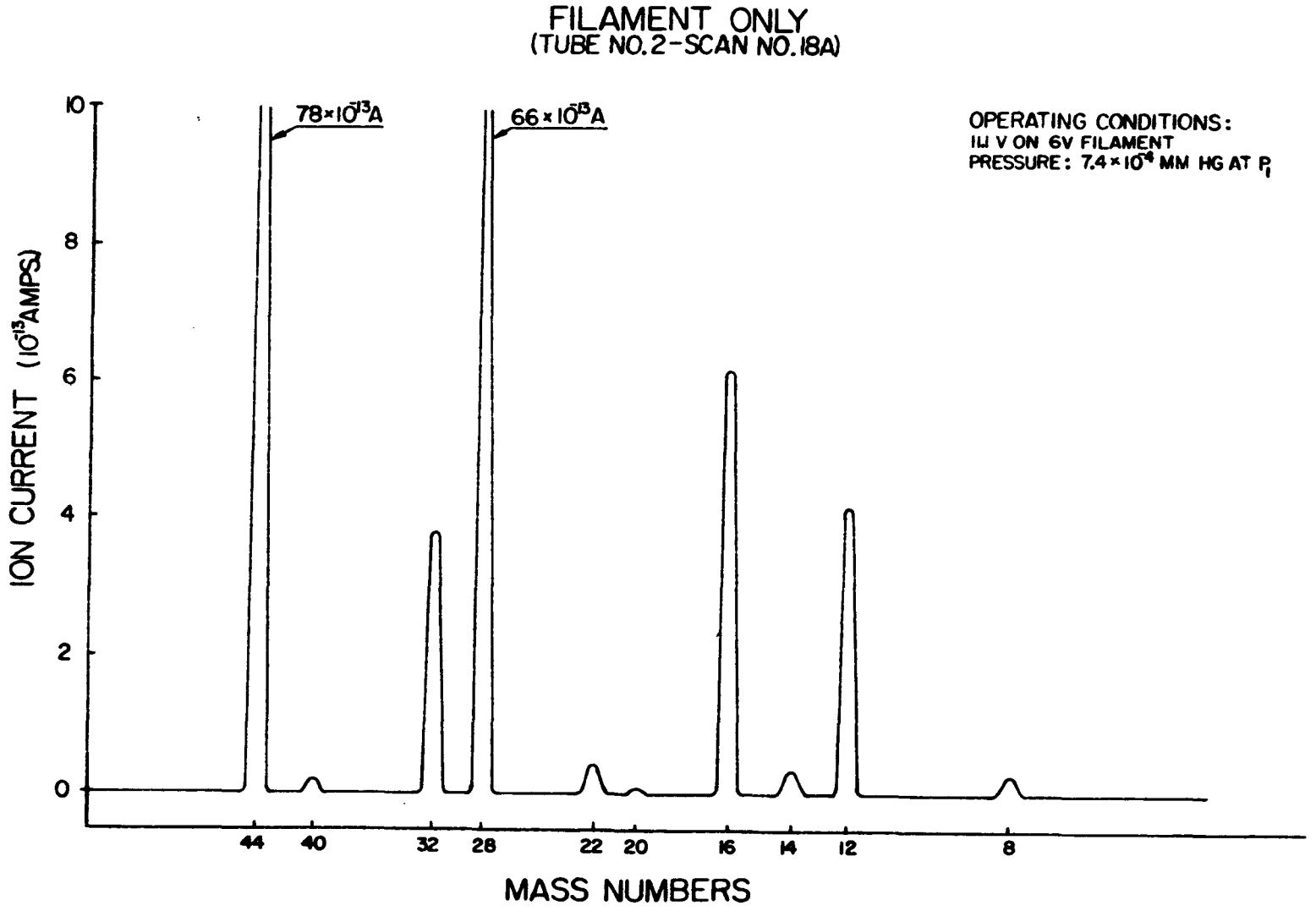
FILAMENT ONLY
(TUBE NO. 2 - SCAN NO. 18A)
78×10⁻¹³A
66×10⁻¹³A
OPERATING CONDITIONS:
111 V ON 6V FILAMENT
PRESSURE: 7.4×10⁻⁶ MM HG AT P₁
ION CURRENT (10⁻¹³AMPS)
10
8
6
4
2
0
44
40
32
28
22
20
16
14
12
8
MASS NUMBERS

FIG. 11

complete cathode breakdown process for Tube No. 3 was performed on the Vacsorb pump.

Figure 12 is a typical scan taken on Tube No. 2 during DC bombarding. As indicated, only H_2 and H_2O are present in quantities above background. The total H_2 evolved during DC bombardment of Tube No. 2 and Tube No. 3 are plotted on Figure No. 13. The totals differ by about 300 micron-liters. Water vapor totals, shown in Figure 14 for the two tubes, differ between Tubes 2 and 3 by a factor of ten (30 vs. 3 micron liters). This difference is probably a result of oxidation or oxygen absorption which may have occurred because of the leak during cathode breakdown of Tube No. 2. The highest temperatures attained by DC bombardment may have desorbed oxygen which then reacted with H_2 from the plates to form H_2O in the same manner as during earlier tests, the deuterium reacted to form D_2O. In fact, the small leak must have existed at the time of the deuterium experiments, also because the nitrogen level was fairly high then too.

For convenience in comparing the quantities of gas evolved from Tube No. 2, the hydrogen (H_2) and water vapor (H_2O) totals have been shown on Figure 9 along with those for CO_2 and CO and for the N_2 which entered through the leak. The CO_2 total does not represent all the CO_2 available, of course, since breakdown was completed on the sorption pump and could not be monitored by the omegatron. Table III tabulates the throughputs and total gas evolved for CO_2, CO, H_2O with the various RF heating, filament voltage and plate current conditions employed on Tube No 2. Figure 15 summarized the throughputs of the gases evolved from Tube No. 3 under various filament voltage and plate current conditions.

DC BOMBARD
(TUBE NO. 2 - SCAN NO. D-7)

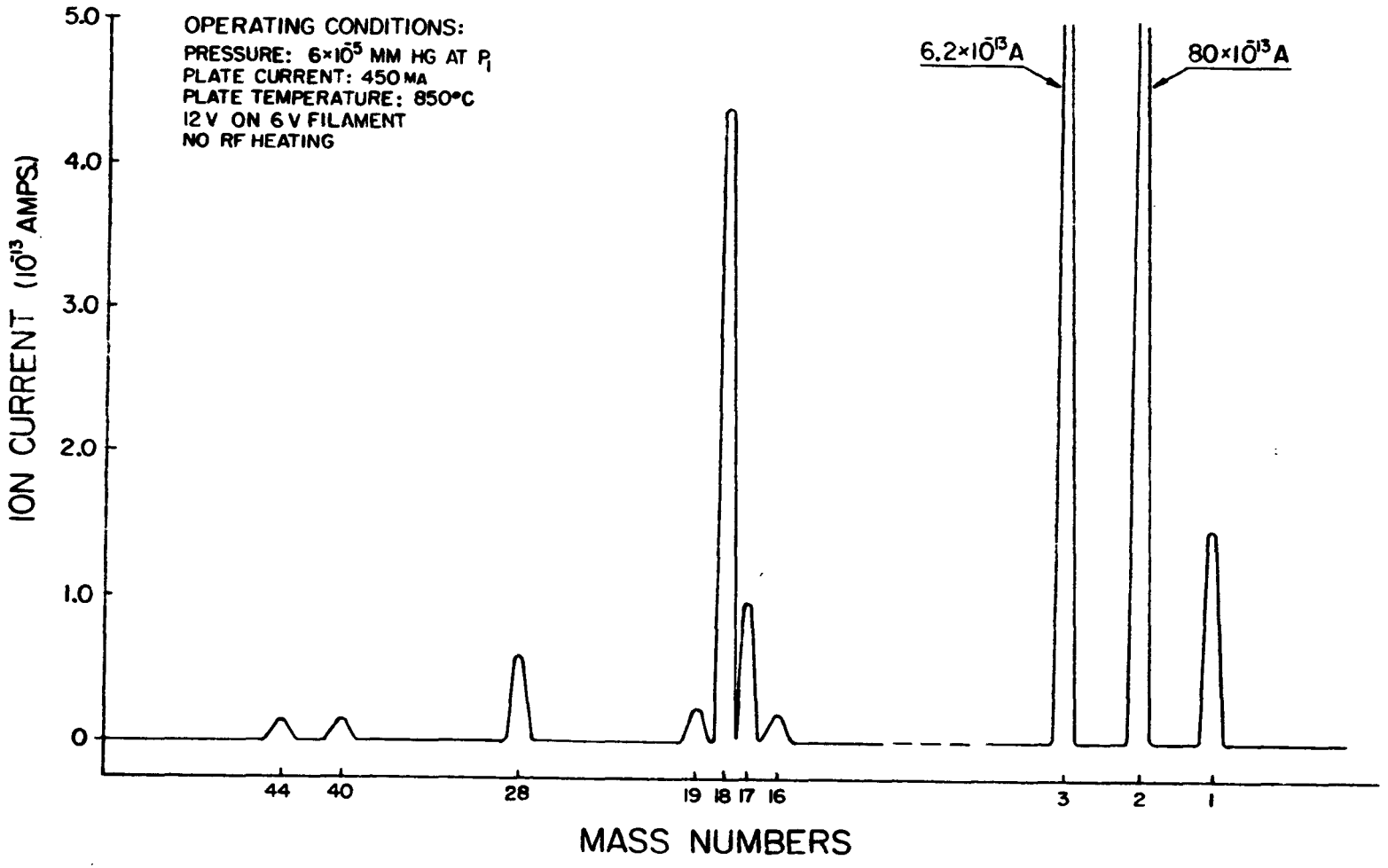

FIG. 12

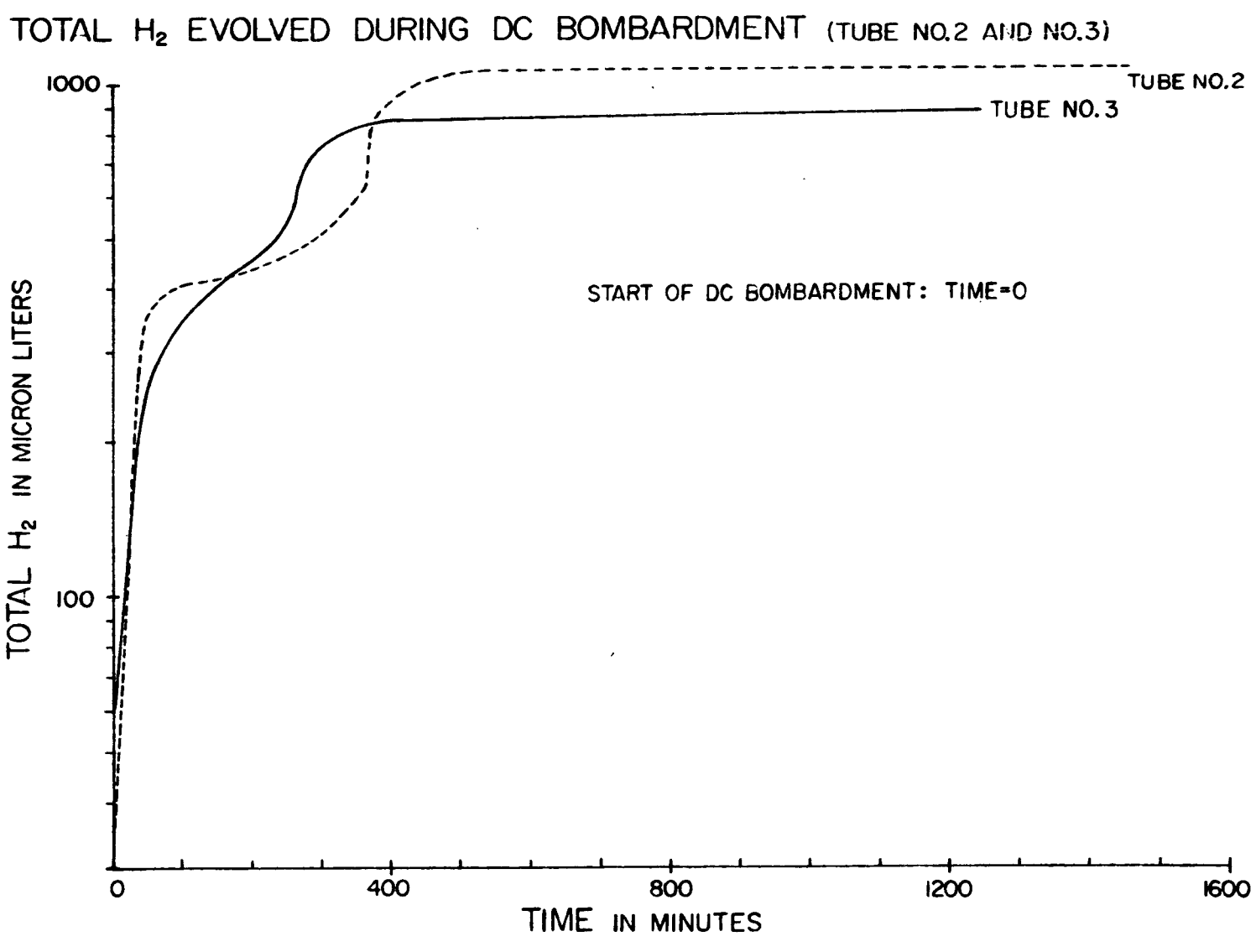
TOTAL H_2 EVOLVED DURING DC BOMBARDMENT (TUBE NO.2 AND NO.3)
1000
100
TOTAL H_2 IN MICRON LITERS
TUBE NO.2
TUBE NO.3
START OF DC BOMBARDMENT: TIME=0
0
400
800
1200
1600
TIME IN MINUTES

FIG. 13

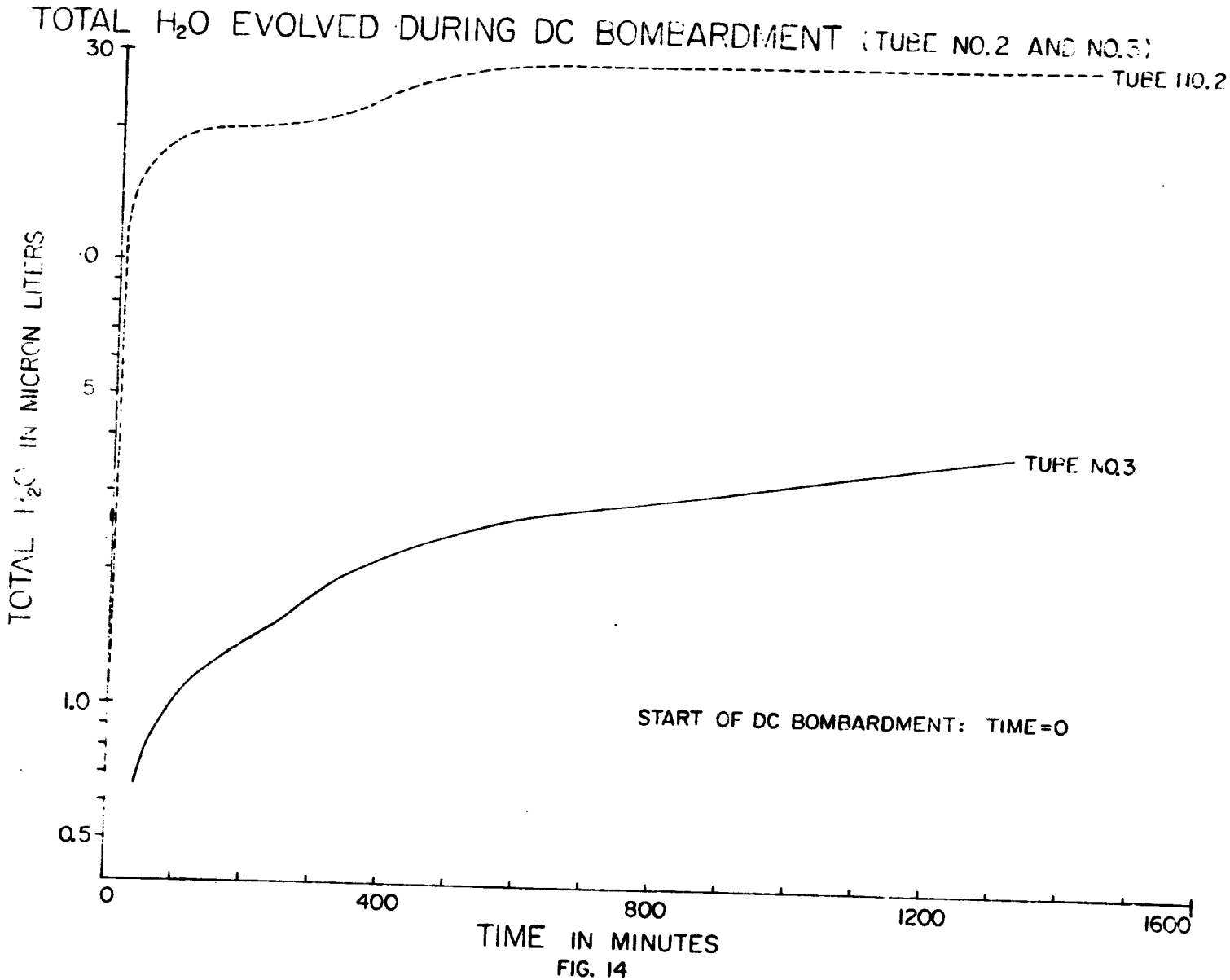
TOTAL H_2O EVOLVED DURING DC BOMBARDMENT (TUBE NO.2 AND NO.3)
TUBE NO.2
TUBE NO.3
START OF DC BOMBARDMENT: TIME=0
TOTAL H_2O IN MICRON LITERS
30
0
5
1.0
0.5
0
400
800
1200
1600
TIME IN MINUTES

FIG. 14

THROUGHPUT VS. TIME
(TUBE NO. 3)

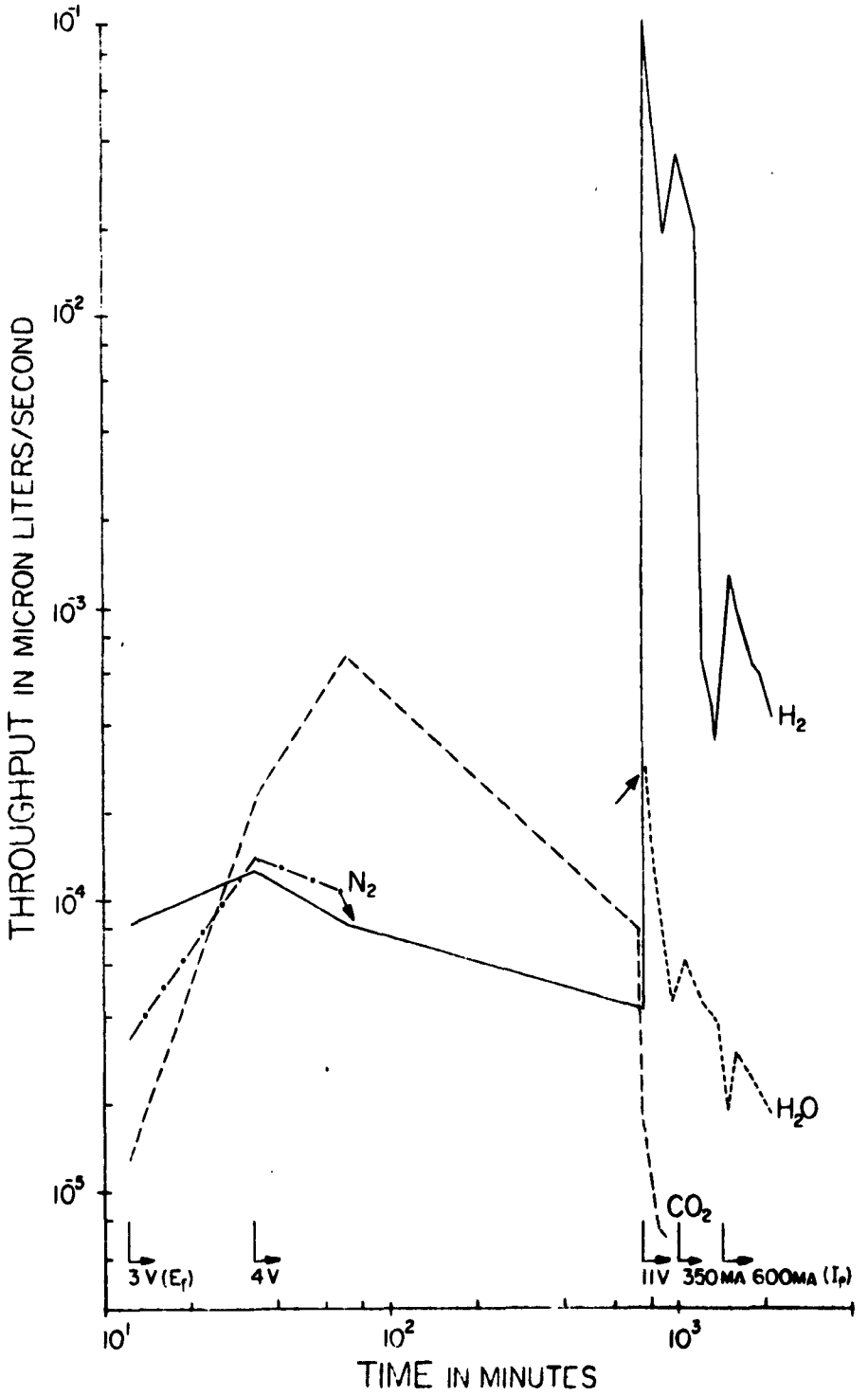

FIG. 15

TABLE III

Elapsed Time	Fil V.	RF	IP	Throughput in ul/s $CO_2 \times 10^3$	$CO \times 10^3$	$H_2 \times 10^3$	$H_2O \times 10^3$	Total ul CO_2	CO	H_2	H_2O	Cumulative Totals CO_2	CO	H_2	H_2O
5	0	.5	0		.048										
15	0	.5	0		.048				.029				.03		
18	0	1.0	0		.052				.009				.04		
23	0	1.0	0	.013	.053				.016				.05		
28	0	1.3	0	.013	.46			.0039	.15				.20		
38	0	1.7	0	.068	12.0		.097	.0246	4.5			.029	4.7		
48	0	1.7	0	.110	8.7		.11	.053	6.0		.60	.082	10.7		60
56	0	1.7	0	.120	7.2		.097	.528	3.4		48	.61	14.1		108
66	0	1.7	0	.140	5.6		.064	.078	3.8		48	.69	18.0		156
78	0	1.7	0	.190			.042	.115			389	.80			545
85	0	2.0	0	6.7	12.7		.45	1.428	10.3		103	2.2	28.2		648
95	0	1.0	0	.90		.084	.019	.288			141	2.5			789
103	0	1.0	0	.43			.045	.3216			15	2.8			804
116	0	1.0	0	.22			.025	.2574			27	3.1			831
123	0	1.0	0	.19			.032	.0882			12	3.2			843
151	0	1.0	0	.16			.019	.1156			420	3.3			1263
181	6.0	1.0	0	4.30		.43		1.68				5.0			
188	6.0	1.0	0	39.5				7.56				12.5			
192	7.5	1.0	0	1.9				3.84				16.4			
196	9.0	1.0	0	5.5		.4		.888				17.3			
202	10.5	1.0	0	8.0				2.448				19.7			
217	12.0	1.0	0	18.0			.064	11.7			166	31.4			1429
225	12.0	1.0	0	19.0			.064	8.64			31	40.1			1460
240	12.0	1.0	0	23.0			.064	18.9			58	59.0			1518
258	12.0	1.0	0	23.0			.042	24.84			57	83.8			1575
378	0	0	0	14.0				13.2				46.4			
398	0	0	0												
414	0	1.0	0												
423	6.0	1.0	0	.06			.0066	18.9			98	115.9			1673
427	9.0	1.0	0	.74				1.146				117.0			
437	12.0	1.0	0	24.0				22.2				139.2			
					Adjust R.F. Bias										
520	12.0	1.0	0	26.0				124.5				263.7			
552	12.0	1.0	0	13.0				38.4				302.1			

Elapsed Time	Fil V.	RF	IP	Throughput in ul/s $CO_2 \times 10^3$	$CO \times 10^3$	$H_2 \times 10^3$	$H_2O \times 10^3$	Total ul CO_2	CO	H_2	H_2O	Cumulative Totals CO_2	CO	H_2	H_2O
580	12.0	1.0	0	5.0				1.5				304			
587	12.0	0	0	1.0				1.3				305			
733	12.0	0	0	.53				6.7				312			
788	10.5	0	0	1.9				4.0				316			
804	10.5	0	0	3.8				2.8				318			
812	10.5	0	0	4.0		.043		1.9				320			
852	10.5	0	0	4.6				10.3				331			
886	10.5	0	0	4.6				9.4				340			
897	10.6	0	0	5.0				3.2				343			
977	10.8	0	0	6.8				28.3				371			
1018	10.8	0	0	8.6				18.9				390			
1054	10.8	0	0	7.7				17.7				408			
1084	10.8	0	0	8.9				14.9				423			
1154	10.8	0	0	9.3				38.2				461			
1186	10.8	0	0	7.2				15.9				477			
2086	11.0	0	0	6.0				39.6				517			
2090	11.0	0	0	6.0				1.4				518			
2120	11.1	0	0	8.0				54.6				573			
2129	11.1	0	0	8.9				4.5				577			
2176	11.3	0	0	5.5				20.3				597			
2185	11.3	0	0	11.0				4.2				602			
2190	11.3	0	0	13.0				3.6				606			
2232	9.0	0	0	1.5				14.9				620			
2267	9.0	0	0	.87				2.0				622			
2342	9.0	0	0	.74				3.6				626			
2418	9.5	0	0	.91				4.0				630			
2525	9.5	0	0	1.0				6.4				637			
2626	9.5	0	0	.87				5.7				642			
3291	9.0	0	0	.91				3.6				646			
3558	9.5	0	0	1.1				15.6				662			
3818	9.5	0	0	1.0				15.6				677			
3843	8.0	0	1.6	.7				1.3				678			
3864	11.0	0	0	.073			.042	4.8				683			
3888	12.016	0	0	1.1				.008				683			

Elapsed Time	Fil v	RF	IP	$CO_2 \times 10^3$	$CO \times 10^3$	$H_2 \times 10^3$	$H_2O \times 10^3$	CO_2	CO	H_2	H_2O	CO_2	CO	H_2	H_2O
	12.0	0	2.3			1.2	.45								
3905	12.0	0	20	.37			1.4	.612			1.339	684			3.0
3914	12.0	0	30	.47		44	2.5	.227		35.1	1.053	684		35.1	4.1
3992	12.0	0	40	.21		36	1.5	1.60		187.2	9.360	686		222.3	13.4
4170	6.0	0	325			30	.3			352.4	9.630			574.7	23.0
	6.0	0	325												
4218	6.0	0	450	.013		56	.47	1.50		123.8	1.123	687		698.5	24.2
4261	6.0	0	550	.013		50	.74	.034		136.7	1.574	687		835.2	25.7
4331				.006		44		.042		197.4		687		1032.6	
4424	6.0	0	390			4.2	.25			133.9	4.401			1166.5	30.1
4479	6.0	0	400			5.2	.077			15.5	.541			1182.0	30.7
4590	6.0	0	200			.13	.032			17.6	.366			1199.6	31.1
4686	6.0	0	200			.085	.026			.6	.167			1200.2	31.2
4819	6.0	0	200			.085	.051			.68	.303			1200.9	31.5
4837	6.0	0	400			1.5	.038			.85	.48			1201.7	31.6
5059	6.0	0	200			.13	.032			10.9	.465			1212.6	32.0
5072	6.0	0	400			1.3	.013			.55	.21			1213.1	32.1
5352	6.0	0	200			.13	.026			11.76	.336			1224.9	32.4

On Tube No. 3, an attempt was made to determine whether any appreciable "sticking" of gases to orifice walls occurred. To do so, the orifice just before the omegatron, O_{23}, was wrapped with heating tape and raised to about 300°C. The omegatron was scanned before and during the heating to detect any change in gas level, all other conditions being held constant. Since H_2 was the principal gas being evolved during the experiment, the m/e=2 peak was monitored constantly. No change in H_2 level was detected. The experiment will be repeated with other gases during the next period.

It is our intent to submit tubes deemed appropriate for electrical and life tests by the manufacturer. Of the first three tubes only No. 2 may be tested. Tube No. 1 was used for such a variety of experiments that electrical test results could not be meaningful while Tube No. 3 developed a crack along a plate lead probably due to overheating during DC bombardment.

PLANS FOR THE NEXT PERIOD

Having discovered the cause for the anomalous behavior during cathode breakdown, we will now repeat experiments of this nature designed to elucidate the interactions of RF heating, filament heating and high bulb temperatures on the kinds and quantities of gases arising during cathode breakdown. The total CO_2 will be determined and compared to the theoretical value and the total H_2 will be compared to determine the range of variability between tubes of the same type. The gettering ability of the zirconium coated plate suggests the interesting possibility of estimating its capacity. By admitting appropriate gases to the system through the variable leak with and without the TUT operating, some measure of the tube's gettering ability may be determined. Such experiments will be initiated if time permits. The possibility that the low conductances of the orifices may modify the composition due to selective "sticking" of some gases to the orifice walls will be given further consideration.

REFERENCES

1. "Electron Tube Materials, Gases and Getters,"

   Final Report, 31 January 1962.
   (Signal Corps Contract No. DA 36-039-sc-87390)

2. Frost, H. B., "Residual Gas Content As a Function of Tube Processing", Symposium on Materials and Electron Device Processing, STP 300, ASTM, 1961.

3. Wagener, J. S., "Properties of Getters in Electron Tubes", Proceedings of the Fourth National Conference on Tube Techniques, New York Un. Press (1958)

PERSONNEL

Contributing to this phase of the contract were the following engineering and scientific personnel:

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Dr. E. I. Doucette

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Air Force Cambridge Research Laboratories, Bedford, Mass. Rept. No. AFCRL 63-35. A QUANTITATIVE STUDY OF THE EVOLUTION OF GASES FROM ELECTRON TUBES AND MATERIALS. Fourth Scientific Report 31 Jan 63, 41p. incl. 15 illus., 3 tables.

Unclassified Report

This is the fourth quarterly report of work aimed at achieving a higher degree of reliability in electron tubes through a better understanding of the kinds and quantities of gases which occur in these tubes. A bakeable, ultra-clean high vacuum system which incorporates an omegatron mass spectrometer has been constructed for conducting these investigations. During this period, several Amperex Type 5894 tubes have been degassed by various schedules. The pumping speed of the ion pump was estimated for methane gas. Several difficulties with heating and operating the tube under test were resolved. The principal gas evolved during DC bombardment of this tube is hydrogen.

1. Electron tube
2. Gases, analysis
3. Mass spectrometers
- I. OAR Project 4619 Task 46190
- II. Contract AF 19(628)-326
- III. E. I. Doucette Associates, Inc., Chatham, New Jersey
- IV. Grubbs, W.J., Snider, G.H., Scott, F.I.,
- V. In ASTIA collection

Air Force Cambridge Research Laboratories, Bedford, Mass. Rept. No. AFCRL 63-35. A QUANTITATIVE STUDY OF THE EVOLUTION OF GASES FROM ELECTRON TUBES AND MATERIALS. Fourth Scientific Report 31 Jan 63, 41p. incl. 15 illus., 3 tables.

Unclassified Report

This is the fourth quarterly report of work aimed at achieving a higher degree of reliability in electron tubes through a better understanding of the kinds and quantities of gases which occur in these tubes. A bakeable, ultra-clean high vacuum system which incorporates an omegatron mass spectrometer has been constructed for conducting these investigations. During this period, several Amperex Type 5894 tubes have been degassed by various schedules. The pumping speed of the ion pump was estimated for methane gas. Several difficulties with heating and operating the tube under test were resolved. The principal gas evolved during DC bombardment of this tube is hydrogen.

1. Electron tube
2. Gases, analysis
3. Mass spectrometers
- I. OAR Project 4619 Task 46190
- II. Contract AF 19(628)-326
- III. E. I. Doucette Associates, Inc., Chatham, New Jersey
- IV. Grubbs, W.J., Snider, G.H., Scott, F.I.,
- V. In ASTIA collection